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(54) Title: AN ENCAPSULATED PARTICLE HAVING AN IMPROVED COATING LAYER (57) Abstract The present invention relates to an encapsulated particle with at least one coating layer containing an amine oxide. The encapsulated particle of the invention is suitable for use in a cleaning composition, for example, a laundry bar composition.		

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AN ENCAPSULATED PARTICLE HAVING AN IMPROVED COATING LAYER

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FIELD

The present invention relates to an encapsulated particle. Specifically, the
10 present invention relates to an encapsulated particle which is useful in a cleaning composition.

BACKGROUND

A typical cleaning composition contains many types of active ingredients.
15 An active ingredient is a compound which has an "activity" when used to clean a substrate. For example, a cleaning composition can contain as an active ingredient, a bleach which bleaches or whitens a substrate. In another example, a cleaning composition can contain as an active ingredient, an enzyme to catalyze bond cleavage in certain types of soils and stains. Thus, in the above
20 examples, a bleach has a bleaching activity, and an enzyme has an enzymatic activity.

However, an active ingredient can be very sensitive to certain destabilizing conditions, such as environmental, processing, and storage conditions. Destabilizing conditions include, for example, exposure to water (i.e., liquid
25 water, humidity, and moisture), temperature changes, excessive heat and cold, the presence of sunlight, and the presence of inherently incompatible compounds. When an active ingredient becomes destabilized, its activity in the composition decreases.

In order to protect an active ingredient from conditions which may
30 destabilize it, it is known to cover the active ingredient with a coating layer so as to form an encapsulated particle. A typical coating layer used to protect an active ingredient contains an anionic surfactant, such as a fatty acid or a soap.

An encapsulated particle may be formed either by spraying the active
ingredient with a hot molten liquid anionic surfactant, or by spraying the active
35 ingredient with an aqueous anionic surfactant solution. However, both of these

methods have drawbacks. When a hot molten liquid anionic surfactant is applied to an active ingredient, the high temperature of the liquid may destabilize the active ingredient and reduce its activity in the composition.

Although an aqueous anionic surfactant solution may be sprayed onto an active ingredient at a lower temperature, this process is also problematic. A typical aqueous anionic surfactant solution is a viscous solution. A viscous solution is difficult to spray onto an active ingredient, because it can easily clog the spraying mechanism. A viscous solution may also form an uneven coating layer on the active ingredient, which is usually undesirable. While a less concentrated, and therefore less viscous aqueous anionic surfactant solution may be sprayed onto a particle, spraying of such a dilute solution may require, for example, several coating steps in order to protect the active ingredient. Multiple coating steps significantly increase the cost of producing the encapsulated particle. This consequently increases the cost of producing the cleaning composition.

Because water destabilizes many types of active ingredients, the typical coating layer is specifically intended to protect the particle from water. However, because a cleaning composition is often used in aqueous solution, a technical paradox arises: the coating layer should protect the active ingredient from water encountered during storage and processing, and yet the coating layer must not inhibit the activity of the active ingredient, when it is used in solution.

Accordingly, the need remains for a coating layer which protects the active ingredient from water encountered during storage and processing, yet allows the active ingredient to deliver its benefit when used in a cleaning function.

SUMMARY

In accordance with the present invention, it has now been found that an improved coating layer can protect the stability of an active ingredient during storage, and yet allow the active ingredient to deliver its benefit during use. The present invention relates to an encapsulated particle containing an active ingredient coated with at least one coating layer containing an amine oxide.

These and other features, aspects, and advantages of the present invention will become evident to those skilled in the art from a reading of the present disclosure with the appended claims.

DETAILED DESCRIPTION

In accordance with the present invention it has been found that an encapsulated particle containing at least one coating layer containing an amine oxide can provide surprising protection for an active ingredient from destabilizing conditions. Therefore, the active ingredient within the encapsulated particle has improved stability and a correspondingly greater activity when used in a cleaning composition. The improved coating layer of the invention can also be coated onto an active ingredient at a lower temperature, thereby reducing the chances that the coating step itself will destabilize the active ingredient. Furthermore, the improved coating layer of the invention allows a more concentrated solution to be applied to the active ingredient, thereby simplifying the coating process.

All percentages, ratios and proportions herein are by weight, unless otherwise specified. Furthermore, all percentages herein are by weight of the coating layer, unless otherwise specified. All temperatures are in degrees Celsius (°C) unless otherwise specified. All documents cited are incorporated herein by reference.

As used herein, the term "alkyl" means a hydrocarbyl moiety which is straight or branched, saturated or unsaturated. Unless otherwise specified, alkyl moieties are preferably saturated or unsaturated with double bonds, preferably with one or two double bonds. Included in the term "alkyl" is the alkyl portion of acyl groups.

The term "coconut oil" is used herein in connection with materials with fatty acid mixtures which typically are linear and have an approximate carbon chain length distribution of about 8% C₈, 7% C₁₀, 48% C₁₂, 17% C₁₄, 9% C₁₆, 2% C₁₈, 7% oleic, and 2% linoleic (the first six fatty acids listed being saturated). Other sources having similar carbon chain length distribution in their fatty acids, such as palm kernel oil and babassu oil, are included within the term coconut oil.

The term "tallow" is used herein in connection with materials with fatty acid mixtures which are typically linear and have an approximate carbon chain length distribution of 2% C₁₄, 29% C₁₆, 23% C₁₈, 2% palmitoleic, 41% oleic, and 3% linoleic (the first three fatty acids listed are saturated). Other mixtures with similar distribution, such as those from palm oil and those derived from various animal tallow and lard, are also included within the term "tallow." The tallow can also be hardened (i.e., hydrogenated) to convert part or all of the unsaturated fatty acid moieties to saturated fatty acid moieties.

The current invention provides an encapsulated particle which, for example, affords greater protection to the active ingredient from water destabilization, has greater resistance to shear forces and temperature changes, and is easier to produce. As is explained below, these surprising benefits are the result of the incorporation of an amine oxide, either alone, or in conjunction with an adjunct coating material, in the improved coating layer of the invention.

A particle which is encapsulated with the improved coating layer of the present invention has improved stability even in the presence of water. Without intending to be limited by theory, it is believed that a coating layer containing an amine oxide can significantly reduce destabilization of the active ingredient caused by water. Because an amine oxide is very hydrophilic, it is believed that it forms an extremely hydrophilic layer which absorbs water before it reaches and destabilizes the active ingredient. The present invention recognizes that the amount of water encountered during storage is typically much less than that encountered when a cleaning composition is used in solution. Accordingly, the hydrophilic nature of the amine oxide protects the active ingredient from degradation caused by water encountered during storage, while remaining soluble in solution.

The improved coating layer of the invention is also surprisingly elastic, and protects the active ingredient from cracking caused by temperature changes. Without intending to be limited by theory, it is believed that temperature changes can cause an encapsulated particle's active ingredient and coating layer to expand and contract at different rates. This may cause a non-elastic coating layer to crack, exposing the active ingredient to destabilizing conditions. However, an elastic coating layer, as provided by the current invention, is able to expand and contract with the active ingredient, and may therefore resist cracking. For example, when the coating layer contains an amine oxide and an anionic surfactant as an adjunct coating material, the amine oxide plasticizes the coating layer by decreasing the crystallinity of the coating layer. Accordingly, the surprising elasticity of the improved coating layer of the invention protects an active ingredient from temperature changes.

The surprisingly elastic coating layer of the invention also protects the active ingredient from cracking caused by shear forces. Shear forces may cause a coating layer to crack, as they pull and stretch the encapsulated particle. An elastic coating layer deforms, without cracking. Therefore, an elastic coating

layer provides greater resistance to cracking, and improved protection for an active ingredient. Furthermore, such an encapsulated particle maintains the activity of the active ingredient in a wider range of compositions.

An encapsulated particle having an elastic coating layer which deforms, but does not crack when subjected to shear forces, is particularly useful for addition to a laundry bar composition. A typical laundry bar production process subjects the laundry bar composition to shear forces as the composition is mechanically worked. These processing conditions tend to destabilize an active ingredient such as a bleach or an enzyme, unless special process steps are taken. These special process steps are typically expensive, in terms of capital outlays for machinery, slower throughput, etc. However, an elastic coating layer can protect an active ingredient, even from typical laundry bar processing conditions. Thus, the current invention allows an active ingredient to be incorporated into a laundry bar composition, without having to resort to expensive processing changes.

Another advantage of the present invention is that the improved coating layer can be sprayed onto an active ingredient at a lower temperature. For example, if the adjunct coating material is an anionic surfactant or a nonionic surfactant, the addition of even a small amount of an amine oxide lowers the melting point and viscosity of the adjunct coating material. An amine oxide has a relatively large radius of hydration around the hydrophilic head group. Although not wanting to be limited by theory, it is believed that the large radius of hydration disrupts the crystal lattice of the solidified surfactant, thereby reducing the crystal lattice bonding strength. Because this results in a lowered surfactant melting temperature, the molten coating layer can be applied to the active ingredient at a lower temperature. A coating layer which is applied at a lower temperature is less likely to destabilize the active ingredient. The present invention therefore allows the improved coating layer to be applied more easily, and under temperature conditions which are less likely to destabilize the active material.

A significant benefit is also seen when the coating layer is to be applied as an aqueous solution, particularly when the aqueous solution contains an adjunct coating material such as a nonionic surfactant or an anionic surfactant. The addition of an amine oxide to such a solution can decrease the viscosity, resulting in a solution which is easier to spray onto a particle. Although not intending to be limited by theory, it is believed that the large radius of hydration

around the hydrophilic head group lowers the viscosity by disrupting the homogeneity of liquid phase. This disruption of the liquid phase results in a solution having a lowered viscosity. As a consequence, a more concentrated surfactant solution may be prepared for any given viscosity. Spraying a more concentrated solution allows the spraying mechanism to substantially coat a particle with a coating layer in a single spraying step. This simplified coating process significantly decreases the production costs associated with producing an encapsulated particle.

The encapsulated particle of the invention may be used alone, or may be added to another composition, for example, a cleaning composition. In a preferred embodiment, the encapsulated particle is added to a detergent composition. More preferably, the encapsulated particle is added to a laundry bar composition.

The encapsulated particle of the current invention comprises an active ingredient which is coated with at least one coating layer comprising an amine oxide. The improved coating layer of the present invention contains either an amine oxide, or an amine oxide in combination with an adjunct coating material.

Active Ingredient

The active ingredient useful in the present invention has an activity when used in a cleaning composition. This activity is a cleaning activity with respect to a substrate, for example, a bleach has a bleaching activity. Accordingly, a preferred active ingredient includes a bleach, a bleach activator, an enzyme, a perfume, a polymer, a brightener, and mixtures thereof. The active ingredient useful herein comprises a single active ingredient, or a plurality of active ingredients.

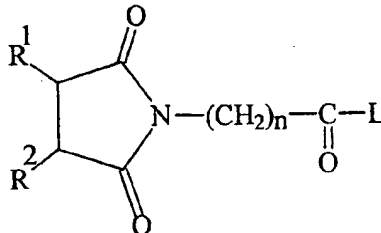
The bleach useful herein has a bleaching activity when used in a cleaning composition, and includes an oxygen bleach, a reducing bleach, a chlorine bleach, a photobleach, a bleach activator, and mixtures thereof. The oxygen bleach useful in the present invention may be any of the oxidizing agents known for laundry, hard surface cleaning, automatic dishwashing or denture cleaning purposes. The oxygen bleach may include, for example, a peroxygen compound, a peracid, an enzymatic source of hydrogen peroxide, and mixtures thereof. A hypohalite such as a chlorine bleach like hypochlorite, may also be used herein. A preferred bleach includes a percarbonate, a perborate, a peracid,

hydrogen peroxide, an oxidase, a persalt, and mixtures thereof. Preferably, the bleach is in solid, particulate, form. Moreover, it is preferred that the bleach particle be highly soluble in water.

The bleach activator useful herein has an activity which increases or promotes a bleach's bleaching activity, and includes an amide, an imide, an ester, an anhydride, and mixtures thereof. Typically, at least one substituted or unsubstituted acyl moiety is present, covalently connected to a leaving group as in the structure $R-C(O)-L$, where L denotes a leaving group. A preferred bleach activator is a peroxygen bleach activator.

10 Preferred bleach activators include N,N,N'-tetraacetyl ethylene diamine (TAED) or any of its close relatives including the triacetyl or other unsymmetrical derivatives. TAED and the acetylated carbohydrates such as glucose pentaacetate and tetraacetyl xylose are preferred hydrophilic bleach activators. Depending on the application, acetyl triethyl citrate, a liquid, also has some utility,
15 as does phenyl benzoate.

Preferred hydrophobic bleach activators include sodium nonanoyloxybenzene sulfonate (NOBS or SNOBS), substituted amide types described in detail hereinafter, such as activators related to 6-nonylamino-6-oxoperoxycaproic acid (NAPAA) as described in U.S. Patent 4,634,551 to Hardy and Ingram, issued January 6, 1987, and activators related to certain imidoperacid bleaches, for example as described in U.S. Patent 5,061,807 to Gethoffer, et al., issued October 29, 1991 and assigned to Hoechst Aktiengesellschaft of Frankfurt, Germany. Japanese Laid-Open Patent Application (Kokai) No. 4-28799 to Yamada, et al., published January 31, 1992 for example describes a bleaching agent and a bleaching detergent composition comprising an organic peracid precursor described by a general formula and illustrated by compounds which may be summarized more particularly as conforming to the formula:



wherein -L is sodium *p*-phenolsulfonate, R¹ is CH₃ or C₁₂H₂₅ and R² is H. Analogs of these compounds having any of the leaving-groups identified herein and/or having R¹ being linear or branched C₆-C₁₆ are also useful.

Other suitable bleach activators include sodium-4-benzoyloxy benzene sulfonate (SBOBS); sodium-1-methyl-2-benzoyloxy benzene-4-sulphonate; sodium-4-methyl-3-benzoyloxy benzoate (SPCC); trimethyl ammonium toluoyloxy-benzene sulfonate; or sodium 3,5,5-trimethyl hexanoyloxybenzene sulfonate (STHOBS).

The enzyme useful herein has an enzymatic activity, and includes an amylase, a cellulase, a cutinase, a lipase, a peroxidase, a protease, and mixtures thereof. Amylases are particularly suitable for automatic dishwashing purposes. An amylase useful herein includes, for example, -amylases described in GB 1,296,839 to Outtrup H, et al., published November 22, 1972 to Novo Industries A/S of Denmark (hereinafter, "Novo"); RAPIDASE® from International Bio-Synthetics, Inc.; TERMAMYL® from Novo; and FUNGAMYL® from Novo.

Cellulases usable herein include both bacterial and fungal types, preferably having a pH optimum between 5 and 9.5. U.S. 4,435,307, to Barbesgaard, et al., March 6, 1984, discloses suitable fungal cellulases from *Humicola insolens* or *Humicola* strain DSM1800 or a cellulase 212-producing fungus belonging to the genus *Aeromonas*, and cellulase extracted from the hepatopancreas of a marine mollusk, *Dolabella Auricula Solander*. Suitable cellulases are also disclosed in GB-B-2,075,028 to Barbesgaard, et al., issued March 28, 1984; GB-B-2,095,275 to Murata, et al., issued August 7, 1985 and DE-OS-2,247,832 to Horikoshi and Ikeda, issued June 27 1974. CAREZYME® and CELLUZYME® (Novo) are especially useful. See also WO 91/17243 to Hagen, et al., published November 14, 1991.

Cutinase enzymes suitable for use herein are described in WO 88/09367A to Kolattukudy, et al., published December 1, 1988.

Suitable lipase enzymes for detergent usage include those produced by microorganisms of the *Pseudomonas* group, such as *Pseudomonas stutzeri* ATCC 19.154, as disclosed in GB 1,372,034 to Dijk and Berg, published October 30, 1974. See also lipases in Japanese Patent Application 53-20487 to Inugai, published February 24, 1978. This lipase is available from Amano Pharmaceutical Co., Ltd., Nagoya, Japan, under the trade name Lipase P "Amano," or "Amano-P." Other suitable commercial lipases include Amano-CES,

lipases ex *Chromobacter viscosum*, e.g. *Chromobacter viscosum* var. *lipolyticum* NRRLB 3673 from Toyo Jozo Co., Tagata, Japan; *Chromobacter viscosum* lipases from U.S. Biochemical Corp., U.S.A. and Disoynt Co., the Netherlands, and lipases ex *Pseudomonas gladioli*. LIPOLASE®, from Novo, is a preferred
5 lipase for use herein. LIPOLASE® is derived from *Humicola lanuginosa*, see also EP 341,947 to Cornelissen, et al., issued August 31, 1994. Lipase and amylase variants stabilized against peroxidase enzymes are described in WO 94/14951 to Halkier, et al., published July 7, 1994 A to Novo. See also WO 92/05249 to Clausen, et al., published April 2, 1992.

10 Peroxidase enzymes may be used in combination with oxygen sources, e.g., percarbonate, perborate, hydrogen peroxide, etc., for "solution bleaching" or prevention of transfer of dyes or pigments removed from substrates during the wash to other substrates present in the wash solution. Known peroxidases include horseradish peroxidase, ligninase, and haloperoxidases such as chloro-
15 or bromo-peroxidase. Peroxidase-containing detergent compositions are disclosed in WO 89/09813 A to Damhus, et al., published October 19, 1989.

A suitable example of a protease is a subtilisin, which is obtained from particular strains of *B. subtilis* and *B. licheniformis*. One suitable protease is obtained from a strain of *Bacillus*, having maximum activity throughout the pH
20 range of 8-12, developed and sold as ESPERASE® by Novo. Other examples of a suitable protease includes ALCALASE® and SAVINASE® from Novo and MAXATASE® from International Bio-Synthetics, Inc., the Netherlands; as well as Protease A and Protease B as disclosed in EP 130,756 A to Bott, published January 9, 1985. An especially preferred protease, referred to as "Protease D,"
25 as described in U.S. Patent 5,679,630 to A. Baeck, et al, issued October 21, 1997, entitled "Protease-Containing Cleaning Compositions," and U.S. Patent 5,677,272 to C. Ghosh, et al, issued October 14, 1997, entitled "Bleaching Compositions Comprising Protease Enzymes."

Commercially-available enzymes are typically available as an enzyme prill,
30 an enzyme marume, a high-shear granule, or even an already-coated granule. Any of these enzyme forms may be coated by the improved encapsulation coating described herein. For example, a preferred embodiment comprises an enzyme prill which contains an enzyme as the active ingredient, and a filler as an additional particle compound. The enzyme prill is coated by the improved
35 coating layer of the invention to form an encapsulated particle. Preferred

examples of commercially-available enzymes useful herein include Savinase®, sold by Novo Corporation, Maxacal sold by Gist-brocades, Opticlean sold by Solvay-Interlox, Co, and Enzoguard sold by Genencor.

In an embodiment of the invention, the encapsulated particle further
5 comprises an additional particle compound which is combined with the active ingredient before it is coated with the coating layer of the invention. The additional particle compound useful herein includes a filler, an enzyme stabilizer, a binder, an acidity source, and mixtures thereof.

The filler useful herein is typically an inert compound upon which the
10 active ingredient is absorbed. However, the filler useful herein may also serve an additional purpose, such as for example, enhancing particle dissolution or providing a buffering effect. Preferred examples of the filler useful herein includes organic and inorganic materials such as talc, alkali metal carbonates, and mixtures thereof.

15 The enzyme stabilizer useful herein depends upon characteristics such as the enzyme used, and the active ingredient. However, preferred examples of an enzyme stabilizer useful herein includes calcium ion, borates, borate-diols, propylene glycol, short chain carboxylic acids, boronic acids, protease-inhibitors, and mixtures thereof.

20 A binder enhances binding of the coating layer to the active ingredient, by increasing the adhesive force between the coating material and the surface of the active ingredient. Additionally, a binder may lower the coating material's surface tension, resulting in a coating layer which is more uniformly spread onto the surface of the active ingredient. Examples of the binder useful herein
25 includes water, polyethylene glycol, glycerol, surfactants, and mixtures thereof.

The additional particle compound may serve as an acidity source or an alkalinity source to further stabilize the active ingredient. For example, a perborate bleach and certain bleach activators may be degraded by alkalinity. An additional particle compound comprising an acidity source helps stabilize the
30 active ingredient from alkaline hydrolysis and other pH-induced degradation. A preferred acidity source includes a small molecular weight polyprotic acid, such as citric acid, malic acid, maleic acid, and mixtures thereof. A preferred alkalinity source includes alkali metal carbonates, such as sodium carbonate.

In a preferred embodiment, a bleach may be combined with a filler, and
35 then coated by an amine oxide. In another preferred embodiment, a bleach and

a bleach activator are combined with a binder and a surfactant, and then coated by a coating layer comprising an amine oxide and an anionic surfactant.

The active ingredient and the additional particle compound may be combined via, for example, agglomeration, mixing, absorption, mixing in solution, and spray drying. In a preferred embodiment, the additional particle compound serves as a base to absorb the active ingredient (or visa-versa). It is preferred to combine an active ingredient with an additional particle compound, as it can help to further stabilize the active ingredient. In a preferred embodiment, the additional particle compound also promotes the dissolution of the active ingredient when the cleaning composition is used. After the active ingredient and the additional particle compound are combined, they are then coated by the improved coating layer of the invention to form an encapsulated particle.

Although not required, it is preferred that the additional particle compound be water-soluble, non-reactive or compatible with the active, and not hygroscopic. A water-soluble additional particle compound is especially preferred because it can dissolve readily in a wash environment and hence does not hinder the dissolution of the active ingredient.

The active ingredient, the additional particle compound, or the combination thereof can be purchased as a raw material, or can be pre-made by methods described above, or known in the art. A preferred active ingredient, additional particle compound, or combination thereof is from about 100 microns to about 1000 microns, more preferably, from about 200 microns to about 800 microns in diameter, before being coated by the coating layer. It is preferred that the active ingredient, additional particle compound, or combination thereof be roughly spherical in shape, as such shapes are more efficiently coated.

Coating Layer

The coating layer useful herein contains an amine oxide, either alone, or in conjunction with an adjunct coating material. When the coating layer contains both an amine oxide and an adjunct coating material, the amine oxide and the adjunct coating material interact to provide synergistic benefits. The coating layer useful herein comprises from about 0.01% to about 100% amine oxide by weight, preferably from about 0.05% to about 90% amine oxide by weight, more preferably from about 0.1% to about 50% amine oxide by weight, and even more

preferably from about 0.5% to about 30% amine oxide by weight, as measured at the time of coating.

The amine oxide useful in the invention also includes those corresponding to the formula: $RR'R''NO$. A more preferred amine oxide corresponds to the formula: $RR'R''NO \cdot xH_2O \cdot yH_2O_2$, in which R is a primary alkyl group containing 8-24 carbons; R' is methyl, ethyl, or 2-hydroxyethyl; R'' is independently selected from methyl, ethyl, 2-hydroxyethyl, and primary alkyl groups containing 8-24 carbons; x is 0 or 1; y is 1 or 2; and $x + y \leq 2$. Also useful herein are modified polyethyleneimine polymers having a polyamine backbone and an amine oxide moiety.

A specific preferred example of a suitable amine oxide for use herein includes, but is not limited to, dodecyldimethyl amine oxide, didecyldimethyl amine oxide, tridecyldimethyl amine oxide, tetradecyldimethyl amine oxide, N-tetracosyldimethyl amine oxide, pentadecyldimethyl amine oxide, hexadecyldimethyl amine oxide, heptadecyldimethyl amine oxide, octadecyldimethyl amine oxide, dodecyldiethyl amine oxide, N-decyl-N-dodecylethyl amine oxide, N-tetradecyl-N-ethylmethyl amine oxide, N-tetradecyl-N-ethyl-2-hydroxyethyl amine oxide, N-docosyl-N-2-hydroxyethylmethyl amine oxide, N,N-ditetradecyl-2-hydroxyethylamine oxide, N-hexadecyldi-2-hydroxyethylamine oxide, hexadecyldiethyl amine oxide, octadecyldiethyl amine oxide, N,N-dieicosylethyl amine oxide, dodecyldipropyl amine oxide, tetradecyldipropyl amine oxide, hexadecyldipropyl amine oxide, octadecyldipropyl amine oxide, dodecyldibutyl amine oxide, tetradecyldibutyl amine oxide, hexadecyldibutyl amine oxide, octadecyldibutyl amine oxide, dodecylmethylethyl amine oxide, tetradecylethylpropyl amine oxide, hexadecylpropylbutyl amine oxide, octadecylmethylbutyl amine oxide, the corresponding monohydrates, and mixtures thereof. Another preferred amine oxide is ADMOX™, a C₁₄ amine oxide dihydrate. ADMOX™ and other amine oxides useful herein are described in U.S. Patent No. 5,292,955 (Smith and Sauer, issued March 8, 1994).

Amine oxide formulations for use in the present process can be prepared by known and conventional methods. Such methods normally involve the controlled oxidation of tertiary amines to the corresponding amine oxide using a strong oxidizing agent. A preferred oxidizing agent is hydrogen peroxide. A dilute, or preferably concentrated (30% by weight or more), hydrogen peroxide

solution is added in a stoichiometric or greater amount to a liquid solution containing the tertiary amine for conversion thereof to the amine oxide. Reaction rates and amine oxide yields can be improved by incorporation of catalysts and or chelating agents well known in the surfactant art for this particular application.

5 Methods for making amine oxide surfactants are described, for example, in U.S. Patent 3,215,741 (Chadwick, issued November 2, 1965), U.S. Patent 3,223,647 (Drew and Voss, issued December 14, 1965), British Patent 437,566 (issued October 31, 1935), and U.S. Patent 4,565,891 (Correa and Riley, issued July 19, 1984).

10 Also useful herein are amine oxide surfactants made by the oxidation of tertiary amines prepared from mixed alcohols obtainable from coconut oil. Such coconut alkyl amine oxides are preferred from an economic standpoint inasmuch as it is not necessary for the present purposes, to separate the mixed alcohol fractions into their pure components to secure the pure chain length fractions of
15 the amine oxides.

The coating layer can contain, in addition to an amine oxide, an adjunct coating material. The adjunct coating material useful herein includes surfactants, such as amphoteric surfactant, anionic surfactant, nonionic surfactant, zwitterionic surfactant, and mixtures thereof. In a preferred embodiment, the
20 adjunct coating material includes both a nonionic surfactant and an anionic surfactant.

If desired, the conventional nonionic and amphoteric surfactants such as the C₁₂-C₁₈ alkyl ethoxylates ("AE") including the so-called narrow peaked alkyl ethoxylates and C₆-C₁₂ alkyl phenol alkoxyates (especially ethoxylates and
25 mixed ethoxy/propoxy), C₁₂-C₁₈ betaines and sulfobetaines ("sultaines"), and the like, can also be included as an adjunct coating materials in the coating layer. A preferred amphoteric surfactant for use herein includes betaines, glycinates, amino/amido propionates, imidazoline-based amphoterics, and mixtures thereof. A preferred nonionic surfactant for use herein includes alkyl ethoxylates, alkyl
30 substituted with phosphine oxides, sulfoxides, and mixtures thereof.

A preferred anionic surfactant for use herein includes a fatty acid, a soap, alkyl sulfates, sulfonates, and mixtures thereof. A more preferred anionic surfactant useful herein includes the conventional C₁₁-C₁₈ alkyl benzene sulfonates ("LAS"), the primary, branched-chain and random C₁₀-C₂₀ alkyl
35 sulfates ("AS"), the C₁₀-C₁₈ secondary (2,3) alkyl sulfates of the formula

$\text{CH}_3(\text{CH}_2)_x(\text{CHOSO}_3^-\text{M}^+) \text{CH}_3$ and $\text{CH}_3(\text{CH}_2)_y(\text{CHOSO}_3^-\text{M}^+) \text{CH}_2\text{CH}_3$ where x and $(y + 1)$ are integers of at least about 7, preferably at least about 9, and M is a water-solubilizing cation, especially sodium, unsaturated sulfates such as oleyl sulfate, the C_{10} - C_{18} alkyl alkoxy sulfates ("AE_xS"; especially EO 1-7 ethoxy sulfates), C_{10} - C_{18} alkyl alkoxy carboxylates (especially the EO 1-5 ethoxycarboxylates), the C_{10} - C_{18} glycerol ethers, the C_{10} - C_{18} alkyl polyglycosides and their corresponding sulfated polyglycosides, and C_{12} - C_{18} alpha-sulfonated fatty acid esters. The C_{10} - C_{18} N-alkyl polyhydroxy fatty acid amides can also be used. Typical examples include the C_{12} - C_{18} N-methylglucamides. See WO 92/06154 to Cook, et al., published April 16, 1992. Other sugar-derived surfactants include the N-alkoxy polyhydroxy fatty acid amides, such as C_{10} - C_{18} N-(3-methoxypropyl) glucamide. The N-propyl through N-hexyl C_{12} - C_{18} glucamides can be used for low sudsing. The fatty acids useful herein are linear or branched carbon chains containing from about 8 to about 24 carbon atoms, preferably from about 10 to about 20 carbon atoms. The average carbon chain length for the fatty acid or soap is from about 12 to about 18 carbon atoms, preferably from about 14 to about 16 carbon atoms. Preferred fatty acids are obtained from coconut oil, tallow, palm oil (palm stearin oil), palm kernel oil, and mixtures thereof. Fatty acids can be synthetically prepared, for example, by the oxidation of petroleum, or by hydrogenation of carbon monoxide by the Fischer-Tropsch process.

As used herein, "soap" means salts of fatty acids. Preferred soaps are alkali metal soaps, such as sodium and potassium soaps, ammonium soaps, and alkylammonium soaps. The soaps useful herein are preferably obtained from natural sources such as plant or animal esters; non-limiting examples include coconut oil, palm oil, palm kernel oil, olive oil, peanut oil, corn oil, sesame oil, rice bran oil, cottonseed oil, babassu oil, soybean oil, castor oil, tallow, whale oil, fish oil, grease, lard, and mixtures thereof. Alkali metal soaps can be made by direct saponification of fats and oils or by the neutralization of the free fatty acids which are prepared in a separate manufacturing process or prepared just prior to application of the coating layer.

Preferred soap raw materials useful herein are soaps made from mixtures of fatty acids from tallow and coconut oil. Typical mixtures have tallow to coconut fatty acid ratios of 85:15, 80:20, 75:25, 70:30, 50:50 and 0:100; preferred ratios are from about 80:20 to about 0:100.

A preferred soap for use herein are neat soaps made by kettle (batch) or continuous saponification. Neat soaps typically comprise from about 65% to about 75%, preferably from about 67% to about 72%, alkali metal soap; from about 24% to about 34%, preferably from about 27% to about 32%, water; and
5 minor amounts, preferably less than about 1% total, of residual materials and impurities, such as alkali metal chlorides, alkali metal hydroxides, alkali metal carbonates, glycerin, and free fatty acids. Another preferred soap raw material is soap noodles or flakes, which are typically neat soap which has been dried to a water content of from about 10% to about 20%.

10 In addition to surfactants, the adjunct coating material useful herein can also include for example, water, paraffin waxes, mica, petrolatum, cod liver oil, squalene, cellulose and cellulose derivatives, inorganic salts (preferably hydrating salts such as phosphates, sulfates, chlorides), and mixtures thereof. It is also preferred that the adjunct coating material be compatible or miscible with
15 soap, or anionic surfactant, or at least dispersible therein.

Although water sometimes damages the active ingredient, it may be present when the coating layer is applied. For example, when the coating layer is applied as an aqueous coating layer solution. In these cases, the encapsulated particle is typically fluidized and surrounded by air, so that the
20 water evaporates by the end of the coating process. Accordingly, any water present during the coating process typically will not destabilize the active ingredient.

It is preferred that the adjunct coating materials have a melting point at or above the temperatures present during, the laundry bar manufacturing process.
25 However, a temperature which is too high may degrade the active ingredient being coated. It is preferred that the melting point of the adjunct coating material be below 100 °C, preferably between about 40 °C and 90 °C.

The coating layer will typically range from about 1 to about 500, preferably from about 5 to about 100 microns thick. In a preferred embodiment, the coating
30 layer is of a substantially uniform thickness throughout. In a preferred embodiment, the viscosity of the adjunct coating material is less than, or equal to the viscosity than the coating layer to which it is to be added.

In a preferred embodiment, the particle is substantially completely coated with at least one coating layer. In a preferred embodiment, the encapsulated
35 particle contains from about 1 to about 5 coating layers, preferably from about 1

to about 2 coating layers. In a preferred embodiment, the coating layer has a melting point of from about 30 °C to about 90 °C, more preferably from about 50 °C to about 85 °C.

At the time at which the coating layer is applied to the particle, the coating
5 layer comprises from about 1% to about 60%, preferably from about 10% to about 40%, and more preferably from about 15% to about 25% of the total encapsulated particle, by weight.

Cleaning Composition

10 In a preferred embodiment, the encapsulated particle of the current invention is added to a cleaning composition, more preferably to a granular or a solid cleaning composition. The cleaning composition may be applicable to, for example, personal cleansing, hard surface cleaning, fabric laundering, manual or automatic dishwashing, and other cleaning applications.

15 In addition to any surfactant contained in the coating layer as an adjunct coating material, a cleaning composition also typically contains a deterative surfactant, such as a cationic surfactant, anionic surfactant, nonionic surfactant, amphoteric surfactant, zwitterionic surfactant, and mixtures thereof. Any of the surfactants listed above as adjunct coating materials may also be used in a
20 cleaning composition as a deterative surfactant. In addition to their presence in the coating layer, the C₁₀-C₁₈ amine oxides are also useful in the cleaning composition as a deterative surfactant. Mixtures of anionic and nonionic deterative surfactants are especially useful. Other useful deterative surfactants are listed in standard texts. Preferably the cleaning composition comprises, by weight of the
25 final composition, at least about 0.01% of a deterative surfactant; more preferably at least about 0.1%; more preferably at least about 1%; more preferably still, from about 1% to about 55%.

Depending upon its desired use, a cleaning composition may also include a plurality of other ingredients, for example, any of the additional particle
30 compounds described above, an alkoxylated polycarboxylate, a brightener, a builder, a carrier, a chelating agent, a clay soil removal/anti-redeposition agent, a dye or pigment, a dye transfer inhibiting agent, a fabric softener, a hydrotrope, a polymeric soil release agent, a polymeric dispersing agent, a processing aid, a solvent, a suds booster, and a suds suppresser.

In a highly preferred embodiment, the encapsulated particle of the invention is added to a detergent bar composition. A preferred detergent bar composition comprises a deterative surfactant, an encapsulated particle, a builder, and a filler. The encapsulated particle preferably contains as the active ingredient at least one of a bleach, a bleach activator, or an enzyme. The detergent bar composition may further include any of the other ingredients disclosed for cleaning compositions.

Coating Process

The encapsulated particle of the invention may be made by conventional encapsulation processes wherein an active ingredient is coated by a coating layer. A preferred process useful herein for coating the particle is a fluidized bed process, such as the Wurster coating process. In a fluidized bed process, the active ingredient is fluidized, and the coating layer is sprayed onto the active ingredient, as it passes by the sprayer(s). The sprayer(s) may be located on the top, bottom and/or the sides of the fluidizing equipment. Co-current, counter-current, from the side, or a combination of these spraying configurations are available. A bottom spray configuration is preferred, because it typically provides more effective spraying.

The Wurster coating process utilizes an enclosed container which contains both a fluidizing mechanism and a spraying mechanism. When the particles are added to the container, they circulate in a given direction. The coating layer is applied by spraying it onto the particles as they pass by the spraying mechanism. In the Wurster process, the particle passes through the spray in a co-current direction.

The improved coating material of the present invention may be sprayed onto the active ingredient as a molten liquid coating layer, in order to form an encapsulated particle. In such a process, special care needs to be taken to reduce or prevent high temperatures which could destabilize the active ingredient. Many oxygen bleaches, for example, may be destabilized by high temperatures. When coating such an oxygen bleach, the temperature of the molten liquid coating layer should be less than about 100 °C, preferably less than 90 °C to avoid destabilizing the oxygen bleach. However, the melting temperature of the improved coating material is lower than comparable coating materials which lack an amine oxide. Accordingly, the molten liquid coating

material of the current invention may be sprayed onto the active ingredient at a lower temperature, and is therefore less likely to destabilize the active ingredient.

Alternatively, the coating layer may be dissolved in water to form an aqueous coating material solution, and then sprayed onto an active ingredient to form an encapsulated particle. Typically, spraying machinery cannot spray solutions which are above a given viscosity, because a viscous solution will easily clog the spraying mechanism. Solution viscosity is directly related to the surfactant concentration in solution; as concentration increases, so does viscosity. Nonionic and anionic surfactant solutions, and particularly soap solutions, are viscous in solution, and difficult to spray. Accordingly, for a given spraying machinery configuration, this effectively translates into an upper "concentration limit" for the solution to be sprayed. The addition of amine oxide to the surfactant solution lowers the viscosity at a given surfactant concentration. Therefore, by adding an amine oxide, this concentration limit is raised. i.e., for a given viscosity, the addition of amine oxide allows the spraying mechanism to spray a more concentrated aqueous coating material solution onto the particle. Therefore, less spray-on time or residence time is required to effectively coat the active ingredient. This simplifies the encapsulation process, and lowers the production costs to produce the encapsulated particle. Moreover, a shorter residence time is highly advantageous when the active ingredient is sensitive to degradation caused by shear.

Examples of the invention are set forth hereinafter by way of illustration and are not intended to be in any way limiting of the invention.

EXAMPLE 1

Examples of active ingredients and additional particle compounds useful in the current invention include:

TABLE I	1	2	3	4	5	6	7
Perborate	100	-	-	-	-	-	-
Percarbonate	-	90	-	-	-	-	-
NOBS	-	-	80	-	-	-	-
Protease	-	-	-	1	-	-	-
Cellulase	-	-	-	-	10	-	-
Brightener	-	-	-	-	-	50	-
Perfume	-	-	-	-	-	-	1
Filler		5		70	20	50	98
Sodium sulfate			10		70		
Binder		5	5	10			1
Balance* to 100%	100%	100%	100%	100%	100%	100%	100%

5 * minors.

All percentages in Table I are by weight of the (unencapsulated) particle.

Coating layer formulations useful in the present invention include:

TABLE II	A	B	C	D	E	F	G
Amine Oxide: (active basis)							
C14AO (87% act.) ADMOX®	100	10	-	-	-	10	-
C12-14 Liquid AO (from Olefins - 35% act.)	-	-	10	-	10	-	10
C10 AO (Barlox10s®) (-30% act.)	-	-	-	10	-	-	-
Adjunct Coating Materials:							
coconut alkyl sulfate	-	-		10	10	10	10
C11.8 LAS	-	-	10	10	10	10	10
80/20	-	90	80	70	60	50	30

Tallow/Coconut soap							
coconut fatty acid	-	-	-	-	10	10	10
C16-18 AE5	-	-	-	-	-	10	10
Carnauba wax	-	-	-	-	-	-	10
Carboxymethyl cellulose	-	-	-	-	-	-	10
Total:	100 %	100 %	100 %	100 %	100%	100%	100%

All percentages in Table II are by weight of the coating layer.

Coating materials B-G showed lower melting points than the corresponding formulations which lack amine oxide. When in solution, these formulations were also less viscous than a comparable solution which lacked amine oxide. Any particle (1-7) is compatible with any coating layer (A-G). Specific examples of an encapsulated particle include: Coating material Example B is highly preferred. Particles 1 and 2 from Table I are also highly preferred, especially when encapsulated by coating materials A and B from Table II.

EXAMPLE 2

Sodium perborate powder having a mean particle size of 300 mm, was purchased from AtoChem, Inc. An aqueous coating layer solution of 70% soap (C₁₂₋₁₈), 29% water, and 1% amine oxide was prepared. The sodium perborate was coated with the aqueous coating layer solution to form an encapsulated particle.

The Wurster coating process, utilizing a co-current bottom-sprayer, was used to apply a coating layer to the fluidized sodium perborate. The coating layer, when initially sprayed onto the particle, comprises about 20% of the encapsulated particle, by weight.

EXAMPLE 3

Sodium percarbonate powder having a mean particle size of 500 mm, was purchased from Solvay-Interlox, Co. A molten liquid coating layer containing 80% fatty acid, and 20% amine oxide, and a melting temperature of 50 °C was

prepared.. The sodium percarbonate was coated with the molten liquid coating layer to form an encapsulated particle according to the process of Example 2.

EXAMPLE 4

- 5 The encapsulated particle of Example 3 was combined with a granular detergent composition, to form a cleaning composition. The granular detergent composition possessed excellent cleaning and bleaching activity.

EXAMPLE 5

- 10 Savinase® enzyme prills from Novo are encapsulated by a solution of coating material B, from Table II. The weight ratio of enzyme prill to coating material at the time of coating was about 40:1. This encapsulated particle was then added to a laundry bar composition, immediately prior to plodding and extrusion. The laundry bar possessed high enzymatic activity both after
15 extrusion, and after aging for 2 weeks.

WHAT IS CLAIMED IS:

1. An encapsulated particle comprising an active ingredient coated with at least one coating layer comprising an amine oxide.
2. The encapsulated particle of Claim 1, wherein the coating layer comprises from about 0.01% to about 100% amine oxide, by weight.
3. The encapsulated particle of Claim 1, wherein the coating layer further comprises an adjunct coating material selected from the group consisting of amphoteric surfactant, anionic surfactant, nonionic surfactant, zwitterionic surfactant, and mixtures thereof.
4. The encapsulated particle of Claim 1, wherein the active ingredient is selected from the group consisting of a bleach, a bleach activator, an enzyme, a perfume, a polymer, a brightener, and mixtures thereof.
5. The encapsulated particle of Claim 1, wherein the amine oxide corresponds to the formula: $RR'R''NO$.
6. The encapsulated particle of Claim 1, wherein the coating layer is sprayed onto the particle.
7. The encapsulated particle of Claim 1, further comprising an additional particle compound, wherein the additional particle compound and the active ingredient are combined before the active ingredient is coated with the coating layer.
8. A laundry bar composition comprising the encapsulated particle of Claim 1.
9. A detergent composition comprising the encapsulated particle of Claim 1.
10. An encapsulated particle comprising an active ingredient coated with at least one coating layer comprising:
 - A. an amine oxide selected from the group consisting of dodecyldimethyl amine oxide, didecylmethyl amine oxide, tridecyldimethyl amine oxide, tetradecyldi-methyl amine oxide, N-tetracosyldimethyl amine oxide, pentadecyldimethyl amine oxide, hexadecyldimethyl amine oxide, heptadecyldimethyl amine oxide, octadecyldimethyl amine oxide, dodecyldiethyl amine oxide, N-decyl-N-dodecylethyl amine oxide, N-tetradecyl-N-ethylmethyl amine oxide, N-tetradecyl-N-ethyl-2-hydroxyethyl amine oxide, N-docosyl-N-2-hydroxyethylmethyl amine oxide, N,N-ditetradecyl-2-

hydroxyethylamine oxide, N-hexadecyldi-2-hydroxyethylamine
oxide, hexadecyldiethyl amine oxide, octadecyldiethyl amine oxide,
N,N-dieicosylethyl amine oxide, dodecyldipropyl amine oxide,
tetradecyldipropyl amine oxide, hexadecyldipropyl amine oxide,
5 octadecyldipropyl amine oxide, dodecyldibutyl amine oxide,
tetradecyldibutyl amine oxide, hexadecyldibutyl amine oxide,
octadecyldibutyl amine oxide, dodecylmethylethyl amine oxide,
tetradecylethylpropyl amine oxide, hexadecylpro-pylbutyl amine
oxide, octadecylmethylbutyl amine oxide, XXX, the corresponding
10 monohydrates, and mixtures thereof; and
B. an adjunct coating material.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/US 98/19916

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C11D17/00 C11D3/386 C11D1/75 C11D3/50 C11D3/42

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 267 046 A (THE CLOROX COMPANY) 11 May 1988 see page 6, last paragraph - page 7, line 37	1,3,4,9
X	EP 0 157 618 A (PUREX) 9 October 1985 see page 3, paragraph 2 see page 3, last paragraph - page 4, paragraph 1 see page 8, paragraph 3 see page 9, last paragraph - page 10, paragraph 1	1-4,9
A	---	7,10
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Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"C" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

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Van Bellingen, I

INTERNATIONAL SEARCH REPORT

International Application No

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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